

10/541,360

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MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
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10/541,360

=> file reg
COST IN U.S. DOLLARS

SINCE FILE ENTRY	TOTAL SESSION
0.21	0.21

FULL ESTIMATED COST

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*
* The CA roles and document type information have been removed from *
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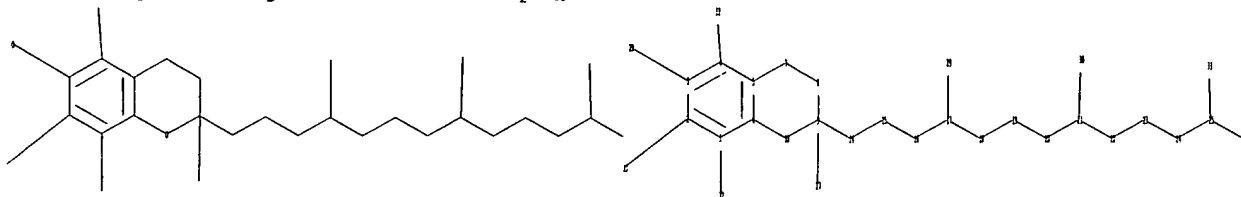
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=>

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10/541,360

chain nodes :
11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31
ring nodes :
1 2 3 4 5 6 7 8 9 10
chain bonds :
1-12 2-13 3-28 4-11 9-14 9-27 14-15 15-16 16-17 17-18 17-29 18-19 19-20
20-21 21-22 21-30 22-23 23-24 24-25 25-26 25-31
ring bonds :
1-2 1-6 2-3 3-4 4-5 5-6 5-7 6-10 7-8 8-9 9-10
exact/norm bonds :
3-28
exact bonds :
1-12 2-13 4-11 5-7 6-10 7-8 8-9 9-10 9-14 9-27 14-15 15-16 16-17 17-18
17-29 18-19 19-20 20-21 21-22 21-30 22-23 23-24 24-25 25-26 25-31
normalized bonds :
1-2 1-6 2-3 3-4 4-5 5-6
isolated ring systems :
containing 1 :

Match level :
1:Atom 2:Atom 3:Atom 4:Atom 5:Atom 6:Atom 7:Atom 8:Atom 9:Atom 10:Atom
11:CLASS 12:CLASS 13:CLASS 14:CLASS 15:CLASS 16:CLASS 17:CLASS 18:CLASS
19:CLASS 20:CLASS 21:CLASS 22:CLASS 23:CLASS 24:CLASS 25:CLASS 26:CLASS
27:CLASS 28:CLASS 29:CLASS 30:CLASS 31:CLASS

L1 STRUCTURE UPLOADED

=> s l1

SAMPLE SEARCH INITIATED 12:05:33 FILE 'REGISTRY'
SAMPLE SCREEN SEARCH COMPLETED - 153 TO ITERATE

100.0% PROCESSED 153 ITERATIONS 50 ANSWERS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE **COMPLETE**
BATCH **COMPLETE**
PROJECTED ITERATIONS: 2318 TO 3802
PROJECTED ANSWERS: 1333 TO 2507

L2 50 SEA SSS SAM L1

=> s l1 ful

FULL SEARCH INITIATED 12:05:39 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 3239 TO ITERATE

100.0% PROCESSED 3239 ITERATIONS 2101 ANSWERS
SEARCH TIME: 00.00.01

L3 2101 SEA SSS FUL L1

=> file caplus

COST IN U.S. DOLLARS

FULL ESTIMATED COST

SINCE FILE
ENTRY
166.94

TOTAL
SESSION
167.15

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FILE LAST UPDATED: 3 Jul 2006 (20060703/ED)

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=> s 13

L4 22026 L3

=> s 14 and acetate

511653 ACETATE

28039 ACETATES

523100 ACETATE

(ACETATE OR ACETATES)

L5 5527 L4 AND ACETATE

=> s 15 and (process or prepar? or synthes? or make or made or method)

2263673 PROCESS

1535806 PROCESSES

3379986 PROCESS

(PROCESS OR PROCESSES)

1655955 PREPAR?

123155 PREP

2160 PREPS

125110 PREP

(PREP OR PREPS)

2014313 PREPD

17 PREPDS

2014325 PREPD

(PREPD OR PREPDS)

122368 PREPG

12 PREPGS

122379 PREPG

(PREPG OR PREPGS)

2719105 PREPN

204810 PREPNS

2873602 PREPN

(PREPN OR PREPNS)

4758712 PREPAR?

(PREPAR? OR PREP OR PREPD OR PREPG OR PREPN)

1542147 SYNTHES?

233459 MAKE

10/541,360

182175 MAKES
403082 MAKE
(MAKE OR MAKES)

1212872 MADE
25 MADES
1212893 MADE
(MADE OR MADES)

3123600 METHOD
1279907 METHODS
4042983 METHOD
(METHOD OR METHODS)

L6 2665 L5 AND (PROCESS OR PREPAR? OR SYNTHES? OR MAKE OR MADE OR METHOD
)

=> s 16 and 2,3,6-trimethylhydroquinone-1-acetate

8787951 2
6611078 3
3735163 6
834 TRIMETHYLHYDROQUINONE
17 TRIMETHYLHYDROQUINONES
840 TRIMETHYLHYDROQUINONE
(TRIMETHYLHYDROQUINONE OR TRIMETHYLHYDROQUINONES)
8723061 1
511653 ACETATE
28039 ACETATES
523100 ACETATE
(ACETATE OR ACETATES)
4 2,3,6-TRIMETHYLHYDROQUINONE-1-ACETATE
(2 (W) 3 (W) 6 (W) TRIMETHYLHYDROQUINONE (W) 1 (W) ACETATE)

L7 3 L6 AND 2,3,6-TRIMETHYLHYDROQUINONE-1-ACETATE

=> s 16 and (2,3,6-trimethylhydroquinone-1-acetate or
1-acetate-2,3,6-trimethylhydroquinone)

8787951 2
6611078 3
3735163 6
834 TRIMETHYLHYDROQUINONE
17 TRIMETHYLHYDROQUINONES
840 TRIMETHYLHYDROQUINONE
(TRIMETHYLHYDROQUINONE OR TRIMETHYLHYDROQUINONES)
8723061 1
511653 ACETATE
28039 ACETATES
523100 ACETATE
(ACETATE OR ACETATES)
4 2,3,6-TRIMETHYLHYDROQUINONE-1-ACETATE
(2 (W) 3 (W) 6 (W) TRIMETHYLHYDROQUINONE (W) 1 (W) ACETATE)

8723061 1
511653 ACETATE
28039 ACETATES
523100 ACETATE
(ACETATE OR ACETATES)
8787951 2
6611078 3
3735163 6
834 TRIMETHYLHYDROQUINONE
17 TRIMETHYLHYDROQUINONES
840 TRIMETHYLHYDROQUINONE
(TRIMETHYLHYDROQUINONE OR TRIMETHYLHYDROQUINONES)
0 1-ACETATE-2,3,6-TRIMETHYLHYDROQUINONE

10/541,360

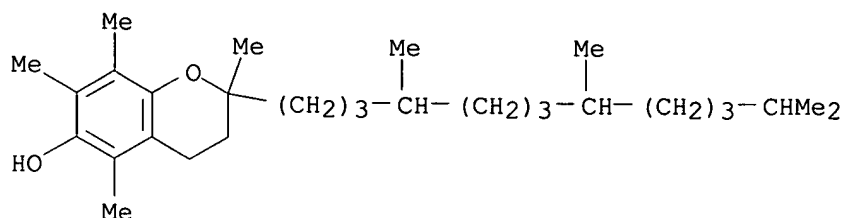
(1(W)ACETATE(W)2(W)3(W)6(W)TRIMETHYLHYDROQUINONE)
L8 3 L6 AND (2,3,6-TRIMETHYLHYDROQUINONE-1-ACETATE OR 1-ACETATE-2,3,6-TRIMETHYLHYDROQUINONE)

=> dup rem 17 18
PROCESSING COMPLETED FOR L7
PROCESSING COMPLETED FOR L8
L9 3 DUP REM L7 L8 (3 DUPLICATES REMOVED)

=> d 19 ibib hitstr abs 1-3

L9 ANSWER 1 OF 3 CAPLUS COPYRIGHT 2006 ACS on STN DUPLICATE 1
ACCESSION NUMBER: 2005:260046 CAPLUS
DOCUMENT NUMBER: 142:336487
TITLE: A new route to α -tocopheryl alkanoates and precursors thereof
INVENTOR(S): Bonrath, Werner; Breuninger, Manfred; Malaise, Gregory; Netscher, Thomas
PATENT ASSIGNEE(S): DSM IP Assets B.V., Neth.
SOURCE: PCT Int. Appl., 36 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
-----	----	-----	-----	-----
WO 2005026142	A2	20050324	WO 2004-EP9749	20040902
WO 2005026142	A3	20050609		
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
EP 1663937	A2	20060607	EP 2004-764709	20040902
R:	AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, FI, RO, CY, TR, BG, CZ, EE, HU, PL, SK			
PRIORITY APPLN. INFO.:			EP 2003-20875	A 20030915
			WO 2004-EP9749	W 20040902
OTHER SOURCE(S):	CASREACT 142:336487; MARPAT 142:336487			
IT 10191-41-ODP, O-acyl				
RL: PNU (Preparation, unclassified); PREP (Preparation)				
(new route to α -tocopheryl alkanoates and precursors thereof via a cross-metathesis)				
RN 10191-41-0 CAPLUS				
CN 2H-1-Benzopyran-6-ol, 3,4-dihydro-2,5,7,8-tetramethyl-2-(4,8,12-trimethyltridecyl)- (9CI) (CA INDEX NAME)				



GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB The present invention is concerned with a novel process for the manufacture of 4-alkanoyloxy-2,3,5-trimethylphenyl (E/Z)-phytyl ethers I, precursors of α -tocopheryl alkanooates II, by cross-metathesis reaction of alkenyl ethers III (R1, R2 = H, C1-5-alkyl, with the proviso that at least one of R1 and R2 \neq H; R3 = C2-5-alkanoyloxy) of 1-alkanoyl-2,3,6-trimethylhydroquinone with 2,6,10,14-tetramethylpentadecene, R4CH:CMe(CH2CH2CH2CHMe)3Me [R4 = H, CH2R5; R5 = OCHO, C2-5-alkanoyloxy, O2CPh, C1-5-alkoxy, OSiR6R7R8; R6, R7, R8 = C1-6-alkyl, Ph] or a phytol derivative, e.g. an ester, an ether or a silyl ether, in the presence of a cross-metathesis catalyst. As the cross metathesis catalyst especially ruthenium metal carbene complexes, e.g., A:RuCl2LL1 [A = CH2, CH-aryl, CHR13, C:C(R13)2, C:CHSi(R14)3, CHCHC(R13)2, C:CHPh, CHCH:CPh2, C:C:CPh2 (aryl = optionally mono- or multiply-substituted C1-5-alkylated or halogenated Ph); G = ethane-1,2-diyl, ethylene-1,2-diyl, cyclohexane-1,2-diyl, 1,2-diphenylethane-1,2-diyl; R9 = ; L1 = PR10R11R12; R10, R11, R12 = C1-8-alkyl, Ph, C6H4Me; R13 = C1-4-alkyl; R14 = C1-6-alkyl, Ph], A:RuCL2L2L3L4 [L2 = L, L1; L3, L4 = pyridyl, 3-bromopyridyl, 3-chloropyridyl], IV [R15, R16 = H; R15R16 = fused benzene ring; R17 = C1-5-alkyl], are suitable which possess (a) ruthenium metal center(s), have an electron count of 16 or 18 and are penta- or hexa- coordinated. Thus, (\pm)-(2E/Z,7R,11R)-I was prepared from 2,3,6-trimethylhydroquinone via O-alkylation with dimethylallyl bromide in THF containing NaH and cross-metathesis with 2,6,10,14-tetramethylpentadecene in PhMe/Me(CH2)11Me containing a catalytic Grubb's ruthenium catalyst type 2 [benzylidenedichloro(N,N-dimesityltetrahydroimidazol-2-yl)(tricyclohexylphosphine)ruthenium]. A further object of the invention is a process for the manufacture of α -tocopheryl alkanooates comprising this reaction.

L9 ANSWER 2 OF 3 CAPLUS COPYRIGHT 2006 ACS on STN DUPLICATE 2

ACCESSION NUMBER: 2004:610127 CAPLUS

DOCUMENT NUMBER: 141:157318

TITLE: Manufacture of α -tocopheryl acetate from the reaction of 2,3,6-trimethylhydroquinone-1-acetate with phytol, iso-phytol or their derivatives in the presence of metal or rare earth metal triflate

INVENTOR(S): Bonrath, Werner; Dittel, Claus; Netscher, Thomas; Pabst, Thomas; Giraudi, Lisa

PATENT ASSIGNEE(S): DSM IP Assets B.V., Neth.

SOURCE: PCT Int. Appl., 21 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

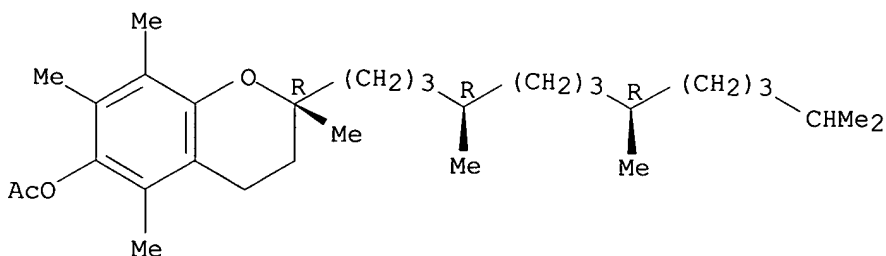
OTHER SOURCE(S) : CASREACT 141:157318; MARPAT 141:157318

IT 52225-20-4P, (all-rac)- α -Tocopheryl acetate
 RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP
 (Preparation)
 (preparation of α -tocopheryl acetate from the
 reaction of trimethylhydroquinone acetate and phytol,
 iso-phytol or their derivs. in the presence of metal or rare earth
 metal triflate)

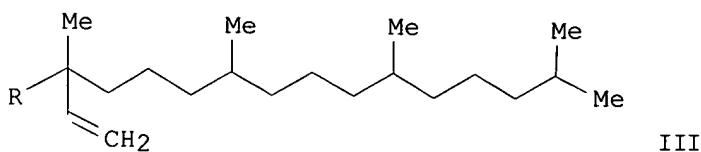
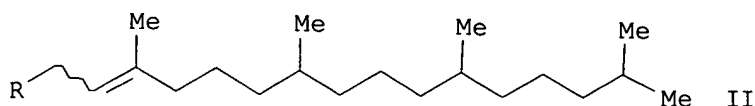
RN 52225-20-4 CAPLUS

CN 2H-1-Benzopyran-6-ol, 3,4-dihydro-2,5,7,8-tetramethyl-2-[(4R,8R)-4,8,12-
 trimethyltridecyl]-, acetate, (2R)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



Page 8



AB The present invention discloses a process for the manufacture of α -tocopheryl acetate (I) by reacting 2,3,6-trimethylhydroquinone-1-acetate with phytol (II; R = OH), iso-phytol (III; R = OH), and their derivs. (R = C2-to C5-alkanoyloxy, benzoyloxy, mesyloxy, benzenesulfonyloxy, tosyloxy) in the presence of a catalyst of the formula $Mn^{+}(R_1SO_3^{-})_n$, wherein Mn^{+} = Ag, Cu, Ga, Hf, rare earth metal cation; n = valence of the cation Mn^{+} ; R_1 = fluorine, C1-8-perfluoroalkyl or perfluoroaryl, and, if required, cyclizing any 3-phytyl-2,5,6-trimethylhydroquinone-1-acetate or a double bond isomer thereof obtained as an intermediate reaction product, to produce I. In the catalyst Mn^{+} is preferably Ag^{+} , Cu^{+} , Ga^{3+} , Sc^{3+} , Lu^{3+} , Ho^{3+} , Tm^{3+} , Yb^{3+} or Hf^{4+} .

L9 ANSWER 3 OF 3 CAPLUS COPYRIGHT 2006 ACS on STN DUPLICATE 3
 ACCESSION NUMBER: 2004:453199 CAPLUS
 DOCUMENT NUMBER: 141:7308
 TITLE: Manufacture of tocopheryl acetate
 INVENTOR(S): Bonrath, Werner; Dittel, Claus; Netscher, Thomas;
 Pabst, Thomas; Schmid, Rudolf
 PATENT ASSIGNEE(S): DSM IP Assets B.V., Neth.
 SOURCE: PCT Int. Appl., 26 pp.
 CODEN: PIXXD2
 DOCUMENT TYPE: Patent
 LANGUAGE: English
 FAMILY ACC. NUM. COUNT: 1
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004046126	A1	20040603	WO 2003-EP10789	20030929
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW			
RW:	GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
AU 2003271655	A1	20040615	AU 2003-271655	20030929
EP 1562929	A1	20050817	EP 2003-753473	20030929
R:	AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK			
CN 1701066	A	20051123	CN 2003-825343	20030929

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JP 2006515280	T2	20060525	JP 2004-552466	20030929
US 2006094886	A1	20060504	US 2005-535604 -	20050519
PRIORITY APPLN. INFO.:			EP 2002-25989	A 20021121
			WO 2003-EP10789	W 20030929

OTHER SOURCE(S): CASREACT 141:7308; MARPAT 141:7308

IT 52225-20-4P 186537-57-5P

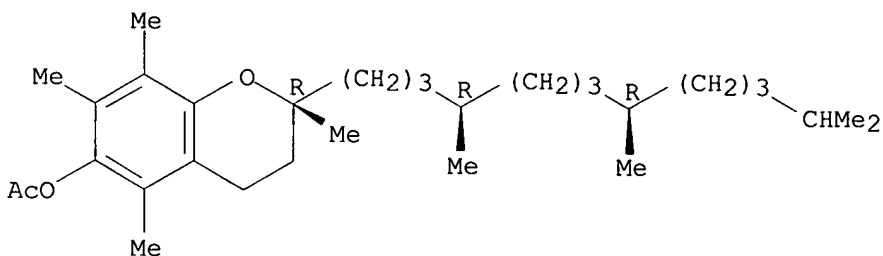
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)

(manufacture of tocopheryl acetate by C-alkylation of 2, 3,6-trimethylhydroquinone-1-acetate with isophytol or phytol in the presence of a sulfur(VI) containing catalyst)

RN 52225-20-4 CAPLUS

CN 2H-1-Benzopyran-6-ol, 3,4-dihydro-2,5,7,8-tetramethyl-2-[(4R,8R)-4,8,12-trimethyltridecyl]-, acetate, (2R)-rel- (9CI) (CA INDEX NAME)

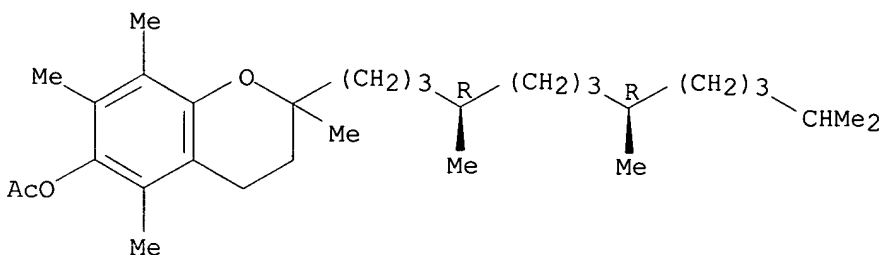
Relative stereochemistry.



RN 186537-57-5 CAPLUS

CN 2H-1-Benzopyran-6-ol, 3,4-dihydro-2,5,7,8-tetramethyl-2-[(4R,8R)-4,8,12-trimethyltridecyl]-, acetate (9CI) (CA INDEX NAME)

Absolute stereochemistry.



AB A process for the manufacture of 3-phytyl-2,5,6-trimethylhydroquinone-1-acetate, and optionally tocopheryl acetate, by either C-alkylating 2,3,6-trimethylhydroquinone-1-acetate with isophytol or phytol in the presence of a sulfur(VI) containing catalyst of the formula R_1SO_2OH (R_1 = hydroxy, halogen, lower alkyl, halogenated lower alkyl or aryl) in an aprotic organic solvent, or O-alkylating 2,3,6-trimethylhydroquinone-1-acetate with a phytyl halide in a polar aprotic organic solvent in the presence of a base, and subjecting the so-obtained 4-O-phytyl-2,3,6-trimethylhydroquinone-1-acetate to a rearrangement reaction, and in each case optionally submitting the so-obtained 3-phytyl-2,5,6-trimethylhydroquinone-1-acetate to a ring closure reaction to produce tocopheryl acetate. The

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invention also includes the novel compound 3-phytyl-2,5,6-trimethylhydroquinone-1-acetate and certain stereoisomers thereof, and also the further novel compound 4-hydroxy-2,3,6-trimethyl-5-[3-(4,8,12-trimethyltridecyl)-but-3-enyl]phenyl acetate which itself is one of several isomers of 3-phytyl-2,5,6-trimethylhydroquinone-1-acetate formed by isomerization under the influence of heating, e.g. during its distillation as part of the isolation and purification procedure following its manufacture as indicated above. (All-rac)- α -tocopherol, which may be derived from its acetate, is known to be the most active industrially important member of the vitamin E group.

=> log y

COST IN U.S. DOLLARS

SINCE FILE	TOTAL
ENTRY	SESSION
67.76	234.91

FULL ESTIMATED COST

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE	TOTAL
ENTRY	SESSION
-2.25	-2.25

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STN INTERNATIONAL LOGOFF AT 12:10:33 ON 04 JUL 2006